AD-A014 153

ASSESSMENT OF STRENGTH-PROBABILITY-TIME RELATIONSHIPS IN CERAMICS

Edward M. Lenoe, et al

Army Materials and Mechanics Research Center

Prepared for:

Army Materiel Command
Advanced Research Project Agency

June 1975

AMMRC TR 75-13

AD

ASSESSMENT OF STRENGTH-PROBABILITY-TIME RELATIONSHIPS IN CERAMICS

EDWARD M. LENOE and DONALD M. NEAL
MECHANICS RESEARCH LABORATORY

June 1975

Sponsored by
Advanced Research Projects Agency
ARPA Order No. 2181

Approved for public release; distribution unlimited.

ARMY MATERIALS AND MECHANICS RESEARCH CENTER
Watertown, Massachusetts 02172

28

The findings in this report are not to be construed as an official Department of the Army position, unless so designated by other authorized documents.

Mention of any trade names or manufacturers in this report shall not be construed as advertising nor as an official indorsement or approval of such products or companies by the United States Government.

DISPOSITION INSTRUCTIONS

Destroy this report when it is no longer needed.
Do not return it to the originator.

iv-a

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE

READ INSTRUCTIONS BEFORE COMPLETING FORM

1. REPORT NUMBER: AMMRC TR 75-13	2. GOVT ACCESSION NO.
3. RECIPIENT'S CATALOG NUMBER	
4. TITLE (and Subtitle): ASSESSMENT OF STRENGTH-PROBABILITY-TIME RELATIONSHIPS IN CERAMICS	5. TYPE OF REPORT & PERIOD COVERED: 6/1/74 - 6/30/75
	6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s): Edward M. Lenoe and Donald M. Neal	8. CONTRACT OR GRANT NUMBER(s)
9. PERFORMING ORGANIZATION NAME AND ADDRESS: Army Materials and Mechanics Research Center, Watertown, Massachusetts 02172, AMXMR-TM	10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS: ARPA Order No. 2181, Program Element 61101E, Agency Accession: DA OE1698
11. CONTROLLING OFFICE NAME AND ADDRESS: U. S. Army Materiel Command, Alexandria, Virginia 22333	12. REPORT DATE: June 1975
	13. NUMBER OF PAGES: 28
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)	15. SECURITY CLASS. (of this report): Unclassified
	15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Silicon nitride
Fracture mechnaics
Monte Carlo method
Ceramics
Slow crack growth
Life prediction
Probability theories

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

(SEE REVERSE SIDE)

DD FORM 1 JAN 73 1473 EDITION OF 1 NOV 65 IS OBSOLETE

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

Block No. 20

ABSTRACT

In the past few years a number of test procedures have evolved as a result of attempts to observe stable crack growth in ceramics under constant stress conditions. The experimental procedures have included double cantilever, double torsion, in-plane moment, and controlled flaws in beam bending tests. These procedures are briefly reviewed. The available slow crack growth data which has been presented in the literature is utilized to estimate survival times for various stress levels. The computations are completed in several ways: (a) use of strength data obtained at various loading rates; (b) deterministic integration of the equations; and (c) in a Monte Carlo sense, wherein the controlling parameters are assumed to possess realistic variability. The end product of each set of computations is a design stress-survival time relationship and the purpose of this paper is to compare these life estimates and comment on the adequacy of each method.

Detailed examination was made of two probability-based procedures for determining stress-probability-time relationships in ceramics. One of these methods relies on strength measurements made under different strain rates, along with the use of a nomograph constructed with the aid of the experimentally determined strength distributions. This procedure does not account for variability in design operating stress, or inherent fracture behavior of the material and therefore a Monte Carlo technique was also studied. Our motivation was to explore the adequacy of the different methods, to assess the status of properties information, and to establish, if possible, reasonable bounds on the accuracy of these probability-based life estimates. Examination of available data and application of the two techniques led to the conclusion that the procedures are appropriate for order of magnitude estimates, which are generally but not necessarily conservative. It was evident that additional data as well as improved experimental procedures and further analytical studies were required. For these reasons the Monte Carlo method was investigated.

Since the exponents of the crack velocity-stress intensity functions in a power law form are large and typically cover a wide range for ceramics of interest, i.e., $4 < n < 50$, difficulties were encountered in use of numerical simulation procedures. With $n \geq 10$, for example, the resulting life functions were widely distributed. Furthermore, the mean value estimates were highly unstable and for large n did not appear amenable to economical digital simulation. Accordingly, a number of different trial functions, including logarithmic, exponential, and polynomial approximations, were employed to represent subcritical crack behavior. The final function selected was of the form $F = A_o \exp(mK_I)$, where $F = K_I/V$.

This form seems appropriate since log F versus K_I resulted in a linear relationship. The n values were appreciably lower than the power law representation. Application of the Monte Carlo method to lifetime estimates of ceramics provided an error tolerance for K_I and allowed estimates of the probability density function.

In summary, comparisons are made of the various life computation procedures wherein realistic properties variability is treated. The predicted behavior of silicon nitride and glass is studied and adequacy of the various estimating procedures is discussed. (Authors)

FOREWORD

This report covers work done in the period June 1, 1974, to June 1975, under the general title "Brittle Materials Research". The work is sponsored by the Advanced Research Projects Agency under ARPA Order No. 2181. The work was carried out at the Army Materials and Mechanics Research Center, Watertown, Massachusetts, 02172, by the principal investigators, E. M. Lenoe and D. Neal. (Phone: 617-926-1900, Ext. 259 or 303)

CONTENTS

INTRODUCTION

At relatively low temperatures the behavior of ceramics is predominantly a strength-controlled phenomena, and the design of ceramic structures is most appropriately based on statistical considerations of strength. However, at elevated temperatures certain ceramics can fail under stresses much lower than those which would ordinarily cause immediate fracture. This condition occurs when the stress/environment combination can cause slow crack growth from existing flaws. In principle, under such circumstances, knowledge of crack velocity and acceleration under a given loading should permit component lifetime estimates to be made. This crack growth velocity, measured in terms of propagation of crack growth under constant load due to creep behavior in ceramics at high temperatures, is not to be confused with fatigue crack propagation.

Several techniques have been developed to estimate lifetimes of ceramic components under either constant or slowly varying stresses. One deterministic method relies on direct integration of crack velocity versus stress intensity relationships. An alternate method of estimating slow crack growth velocity-stress intensity relationships utilizes strength measurements obtained at varying loading rates. Finally, in an effort to provide a probability basis for ceramic structural life calculations, the Weibull strength distribution has been combined with crack velocity-stress intensity functions.

Current efforts to provide a probability basis for the failure times by introducing Weibull strength distributions must be considered with caution. The derivation of the Weibull distribution is based on the assumption that occurrence of a single inhomogeneity in the elemental volume produces fracture. It is possible to generalize statistical strength distribution in terms of more realistic descriptions of microstructural details. Thus, alternate strength-probability functions can be considered in life estimates. Use of statistical strength distributions is generally not sufficient to place the life estimates on a proper probability basis. Other material properties, such as fracture toughness and crack growth phenomena, also possess inherent variability which must be taken into account.

Slow crack growth velocity measurements can be made in a variety of ways, the most popular being the so-called double torsion, edge-notched plate.[1-3] Constant in-plane rather than out-of-the-plane bending moments can also be applied.[4] More recently, flawed beams,[5] possessing diamond-pyramid-induced defects, have been used to determine the crack velocity-stress intensity function. Each of these test methods yields slightly different parameters, with obvious influence on life estimates. Crack velocity behavior in the majority of structural ceramics can be represented by a power function. Due to the typically large exponent n of this power rule, failure time estimates are extremely sensitive to variability of the input parameters.

1. KIES, J. A. and CLARK, B. J. *Fracture Propagation Rates and Times to Fail Following Proof Stress in Bulk Glass.* In Fracture 1969. P. L. Pratt, Ed., Chapman and Hall, London, 1969, p. 483-491.
2. BEACHEM, C. D., KIES, J. A., and BROWN, B. F. *A Constant K Specimen for Stress Corrosion Cracking Tests.* Materials Research and Standards, v. 11, no. 4, April 1971, p. 30.
3. WILLIAMS, D. P. and EVANS, A. G. *A Simple Method for Studying Slow Crack Growth.* J. Test Eval., v. 1, no. 4, July 1973, p. 264-270.
4. FREIMAN, S. W., MULVILLE, D. R., and MAST, P. W. *Crack Propagation Studies in Brittle Materials.* Journal of Materials Science, v. 8, no. 11, November 1973, p. 1527-1533.
5. PETROVIC, J. J., JACOBSON, L. A., TALTY, P. K., and VASUDEVAN, A. K. *Controlled Surface Flaws in Hot-Pressed Si_3N_4.* To be published in Journal of the American Ceramic Society, v. 58, no. 5-6, May-June 1975.

Therefore it is appropriate to examine the experimental procedures and analytical techniques in some detail. This work was prompted in part by Wilkins'[6] objections to current life estimating procedures which did not account for variability in materials properties and which did not provide a firm theoretical basis for the use of a power rule.

EXPERIMENTAL PROCEDURES FOR SLOW CRACK GROWTH

The configurations shown in the schematic of Figure 1 have been used to observe slow crack growth in ceramics. It is worthwhile to consider these methods in turn and outline the basis of data interpretation for each procedure.

A. Double Torsion

The double torsion (DT) specimen has been widely reported[3,7,8] as suitable for subcritical crack growth. However, the configuration has thus far been subjected only to approximate analysis[3] wherein the specimen is treated as two elastic bars; for configurations with large width-to-thickness ratios the torsional strain is given by

$$\theta \approx y/w_m \approx \frac{6Ta}{Wt^3\mu} \qquad (1)$$

where w_m is the moment arm; T the torsional moment $(P/2)w_m$, P/2 the total load applied to one bar; a the crack length; W/2 the bar width; t the bar thickness; μ the shear modulus of the material. Unless the slenderness ratio is large (> 10), Equation (1) can be in considerable error, as attested by reference to standard texts (Reference 9, p. 273). The following equations are used for specimen analysis. First, the relation between stress intensity K and strain energy release rate for crack extension G:

$$K = (EG)^{1/2} \qquad (2)$$

with E = Young's modulus

Secondly, the relation between specimen compliance and strain energy release rate:

$$G = P^2/2\ (dc/dA) \qquad (3)$$

with P = applied load

c = compliance and

A = crack area.

6. WILKINS, B. J. S. and SIMPSON, I. A. *Errors in Estimating the Minimum Time-to-Failure in Glass.* Journal of the American Ceramic Society, v. 57, no. 11, November 1974, p. 505.
7. EVANS, A. G. *Method of Evaluating the Time-Dependent Failure Characteristics of Brittle Materials and Its Application to Polycrystalline Alumina.* Journal of Materials Science, v. 7, no. 10, October 1972, p. 1137-1146.
8. EVANS, A. G. *Slow Crack Growth in Brittle Materials Under Dynamic Loading Conditions.* International Journal of Fracture. v. 10, no. 2, June 1974, p. 251-259.
9. TIMOSHENKO, S. and GOODIER, J. N. *Theory of Elasticity.* 2nd ed., McGraw-Hill, New York and London, 1951.

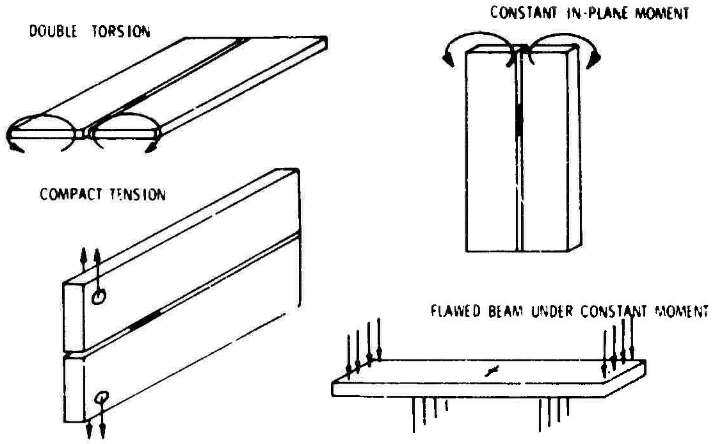


Figure 1. Schematic of Typical Slow Crack Growth Specimens

Assuming a complete through-crack and a crack front which is independent of crack length, then

$$G = P^2/2t_n \ (dc/da) \tag{4}$$

where t_n = plate thickness in crack plane. These equations, together with the approximate expression for specimen compliance

$$c = Y/P = (3w_m{}^2 a)/(Wt^3\mu) \tag{5}$$

lead to the nominal form of stress intensity.

$$K = Pw_m \sqrt{3(1+\nu)/(Wt^3 t_n)} \tag{6}$$

The double torsion specimen was subjected to experimental compliance calibration at room temperature for 4340 steel. The experimental results compared within 9 percent of the predictions of Equation (6). Williams and Evans,[3] however, point out that crack front curvature can lead to significant error in determination of crack velocities if only surface velocities are used. It appears that additional study is required of such curvature effects. Furthermore, a more accurate estimate of stress intensity is required.

B. Constant In-Plane Moment

Rather than apply an edge twisting moment, the edge-notched plate has been also subjected to in-plane moments. Freiman et al.[4] has provided an approximate analysis based on a beam-on elastic foundation model, as well as experimental verification of his strain energy release rate expressions. As in the DT specimen, the results were within 10 percent of the analytical model at ambient temperatures. Crack velocity versus stress intensity data was obtained and compared to previous

studies on soda-lime-glass using the so-called compact tension[10] and double torsion[7] configurations. While the slopes of the resulting relationships are all similar, the curve for DT specimens appears shifted very slightly to the right, whereas the constant moment (CM) results are slightly to the left of those obtained with compact tension test procedures.

C. Compact Tension

This widely used configuration has been subjected to extensive analyses.[11,12] Theory of elasticity solutions, including corrections for finite geometries and various loading modes, are readily available.[13] Unfortunately, Si_3N_4 does not exhibit slow crack growth except at high temperatures (~1800-1900°F). This compounds the difficulties of measuring opening mode displacement and crack growth, and at the time of this writing no slow crack growth data has been reported in the open literature for this configuration.

D. Flawed Beam Under Constant Moment

The cracked beam specimen has been subjected to finite element and other approximate analysis, and estimates can be made for stress intensity factors in a beam under constant moment and containing a semi-elliptical surface flaw.[14-16] The difficulty of developing a natural, sharp crack in ceramics has lead to increasing popularity of an artificially induced flaw.[5] A diamond indenter under prescribed load (Vickers and/or Knoop hardness tester) has been found suitable to introduce small and sharp cracks. Subsequent annealing of the specimen apparently relieves residual stresses so that fracture toughness values are obtained which are identical to those measured using the more acceptable CT specimens.[5]

It is interesting to note that according to rudimentary analysis the stress intensity varies with position along the crack front. Thus if slow crack growth occurs under constant moment, fractography of the failed surfaces can yield a number of crack velocity-stress intensity values from a single test specimen.

It is obvious that the experimental procedures, in general, rely on approximate analysis. Undoubtedly, data interpretation would be improved with a more accurate theoretical basis. This is particularly true of the double torsion configuration. It is also worth mentioning that in Si_3N_4 subcritical crack growth occurs at elevated temperatures where the material has a nonlinear stress-strain curve. The ceramic is capable of increased elongation at its highest usage temperature, which suggests the necessity to account for such nonlinearities.

10. WIEDERHORN, S. M. *Influence of Water Vapor on Crack Propagation in Soda-Lime Glass.* Journal of the American Ceramic Society, v. 50, no. 8, August 1967, p. 407-414.
11. WIEDERHORM, S. M., SHORB, A. M., and MOSES, R. L. *Critical Analysis of the Theory of the Double Cantilever Method of Measuring Fracture - Surface Energies.* Journal of Applied Physics, v. 39, no. 3, 15 February 1968, p. 1569-1573.
12. GUTSHALL, P. L. and GROSS, G. E. *Fracture Energy of Polycrystalline Beryllium Oxide.* Journal of the American Ceramic Society, v. 51, no. 10, October 1968, p. 602.
13. SIH, G. S. *Handbook of Stress Intensity Factors.* Institute of Fracture and Solid Mechanics, Bethlehem, Pa., 1973.
14. MARS, G. R. and SMITH, C. W. *A Study of Local Stresses Near Surface Flaws in Bending Fields.* In Stress Analysis and Growth of Cracks. STP 513, ASTM, Pa., 1972, p. 22-36.
15. CRANDT, A. F., Jr. and SINCLAIR, J. M. *Stress Intensity Factors for Surface Cracks in Bending.* In Stress Analysis and Growth of Cracks, STP 513, ASTM, Pa., 1972, p. 37-58.
16. SHAH, R. C. and KOBAYASHI, A. S. *Stress Intensity Factor for an Elliptical Crack Approaching the Surface of a Plate in Bending.* In Stress Analysis and Growth of Cracks, STP 513, ASTM, Pa., 1972, p. 3-21.

Currently available slow crack growth data for this material has been obtained with either double torsion[17,18] or flawed beam configuration.[19] Kinsman's observations are illustrated in Figures 2 and 3 while Figure 4 compares the crack velocity observations for the different procedures. It is evident that a wide variation in behavior is possible, and the results are dependent on material purity, as well as nominal stress intensity level. Differences in the observed crack velocity versus nominal stress intensity are also exaggerated by deficiencies in theoretical analysis of the test configuration. With the exception of indicated high purity data, the other results of Figure 4 were obtained for the HS-130 version of hot-pressed Si_3N_4. Variations of impurity content between billets may partially account for differences in slope of the data.

ELEMENTARY LIFE ESTIMATING PROCEDURE

Charles[20,21] has proposed and experimentally verified analytical fracture mechanics on models to describe static fatigue and dynamic strength behavior of glass. The analysis uses a proposed power law form of crack velocity versus stress intensity relation using the Griffith criteria. Experimental verification of the proposed relationship relied on several thousand experiments performed on soda-lime-glass rods. It is worthwhile to note that the specimens were subjected to severe surface grinding, which insured sufficient numbers of surface flaws that failure usually occurred at these surface flaws.

In his preliminary discussion Charles discussed restrictions as to the measure of dispersion of the critical initial flaws on similar samples (see Reference 21, p. 1658 and 1659). Such restrictions would tend to select similar distributions of critical flaw sizes for the different groups of specimens. It is also noteworthy that the surface grinding preparation no doubt tended to emphasize fracture related to surface integrity rather than inherent material flaws.

Since those early studies, his concepts have been applied to ceramic materials by a number of investigators to estimate slow crack growth parameters.[22-26]

Using the Griffith equation in the form

$$K_{IC} = Y \sigma_f \sqrt{C_o} \tag{7}$$

17. EVANS. A. G., RUSSELL, L. R., and RICHERSON, D. W. *Slow Crack Growth in Ceramic Materials at Elevated Temperatures.* Metallurgical Transactions A, v. 6A, no. 4, April 1975, p. 707-716.
18. EVANS, A. G. *High-Temperature Slow Crack Growth in Ceramic Materials.* In Ceramics for High Performance Applications, J. J. Burke, A. E. Gorum, and R. N. Katz, Eds., Brook Hill Publishing Company, Chestnut Hill, Mass., 1975, p. 373-396.
19. KINSMAN, K. R. *The Varied Role of Plasticity in the Fracture of Inductile Ceramics.* Ford Motor Company, Scientific Research Staff Publication, Dearborn, Mich., October 1974.
20. CHARLES, R. J. *Static Fatigue in Glass.* Journal of Applied Physics, v. 29, no. 11, November 1958, p. 1958, 1549-1553, 1554-1560.
21. CHARLES, R. J. *Dynamic Fatigue of Glass.* Journal of Applied Physics, v. 29, no. 12, December 1958, p. 1657-1667.
22. WIEDERHORN, S M. *Fracture Surface Energy of Glass.* Journal of the American Ceramic Society, v. 52, no. 2, February 1969, p. 99-105.
23. EVANS, A. G. and WIEDERHORN, S. M. *Proof Testing of Ceramic Materials - An Analytical Basis for Failure Prediction.* International Journal of Fracture, v. 10, no. 3, September 1974, p. 379-392.
24. WIEDERHORN, S. M., EVANS, A. G., and ROBERTS, D. E. *A Fracture Mechanics Study of the Skylab Windows.* In Fracture Mechanics of Ceramics, v. 2, R. C. Bradt, D. P. H. Hasselman, and F. F. Lange, Eds., Plenum Press, New York, 1974, p. 829-841.
25. LINZER, M. and EVANS, A. G. *Failure Prediction in Structural Ceramics Using Acoustic Emission.* Journal of the American Ceramic Society, v. 56, no. 11, November 1973, p. 575-581.
26. DAVIDGE, R. W., McLAREN, J. R., and TAPPIN, G. *Strength-Probability-Time Relationships in Ceramics.* Journal of Materials Science, v. 8, no. 12, December 1973, p. 1699-1705.

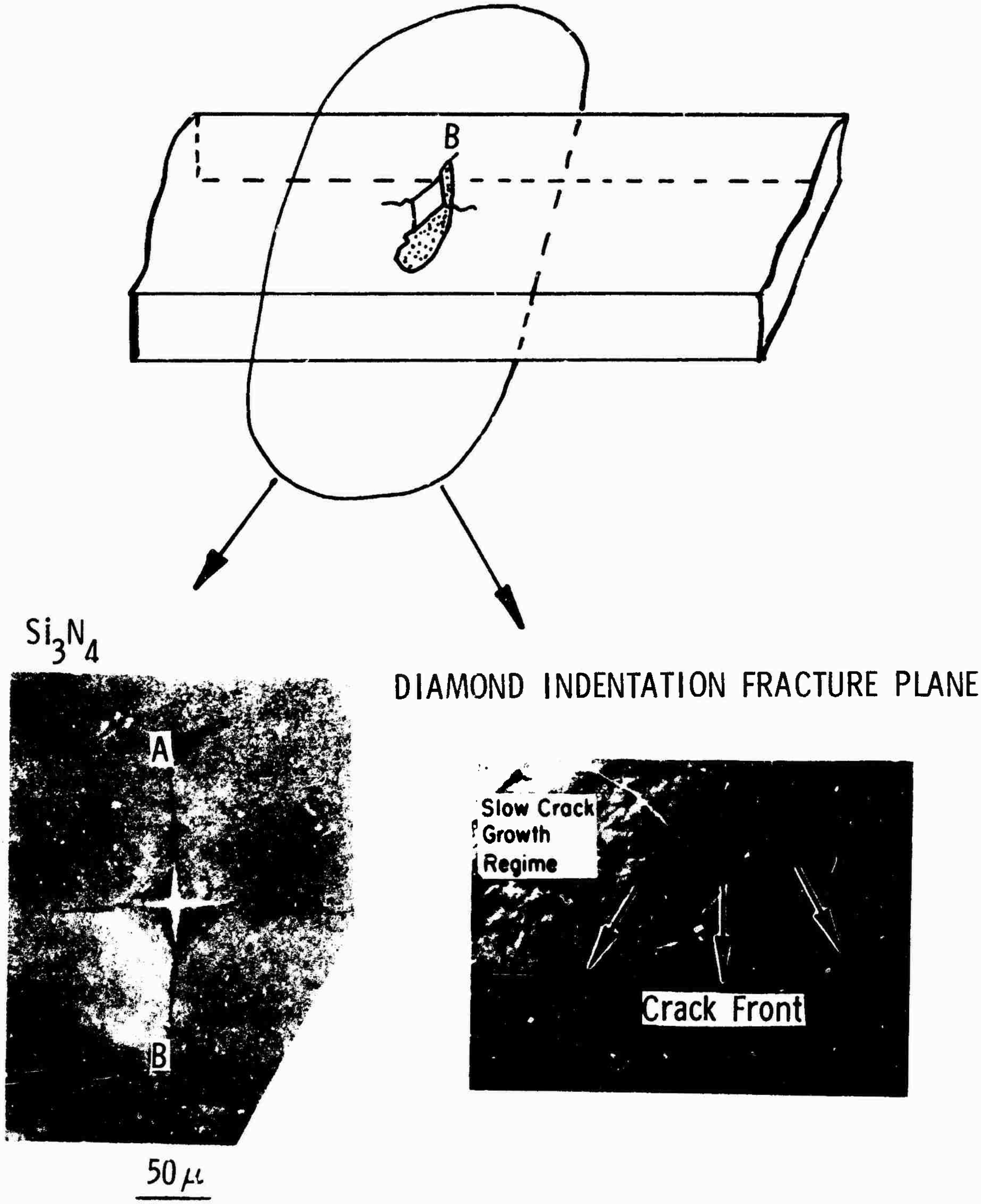
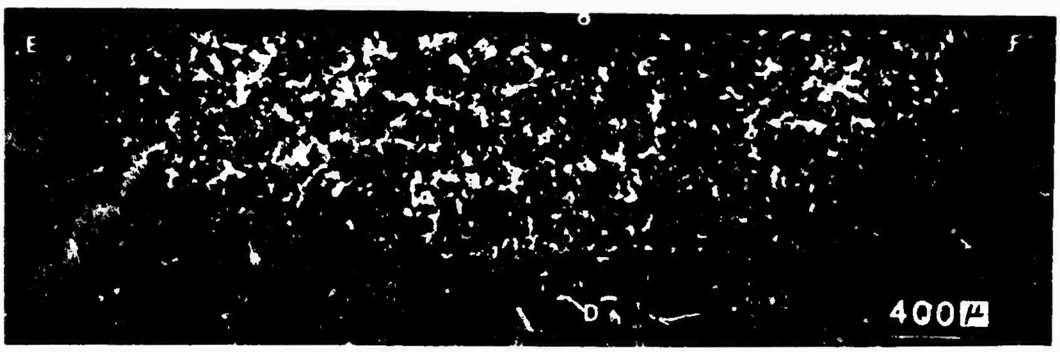


Figure 2. Typical Diamond Pyramid Induced Flaws in Hot-Pressed Silicon Nitride (after Kinsman, Ref. 19)

19-066-904/AMC-75

a. 65.9 ksi
(454.2 MN/m^2)

$\sigma_{A\cdot C} \approx 0.90\ \sigma_F$, 0.5 min



$\sigma_{A\cdot C} \approx 0.73\ \sigma_F$, 1.4 min

b. 53.4 ksi (368.4 MN/m^2)

c. 45.4 ksi (312.9 MN/m^2)
Interrupted Test

$\sigma_{A\cdot C} \approx 0.62\ \sigma_F$, 40 min

Figure 3. Slow Crack Growth Observations in Flawed Beams, Hot-Pressed Silicon Nitride (after Kinsman, Ref. 19)

19-066-903/AMC-75

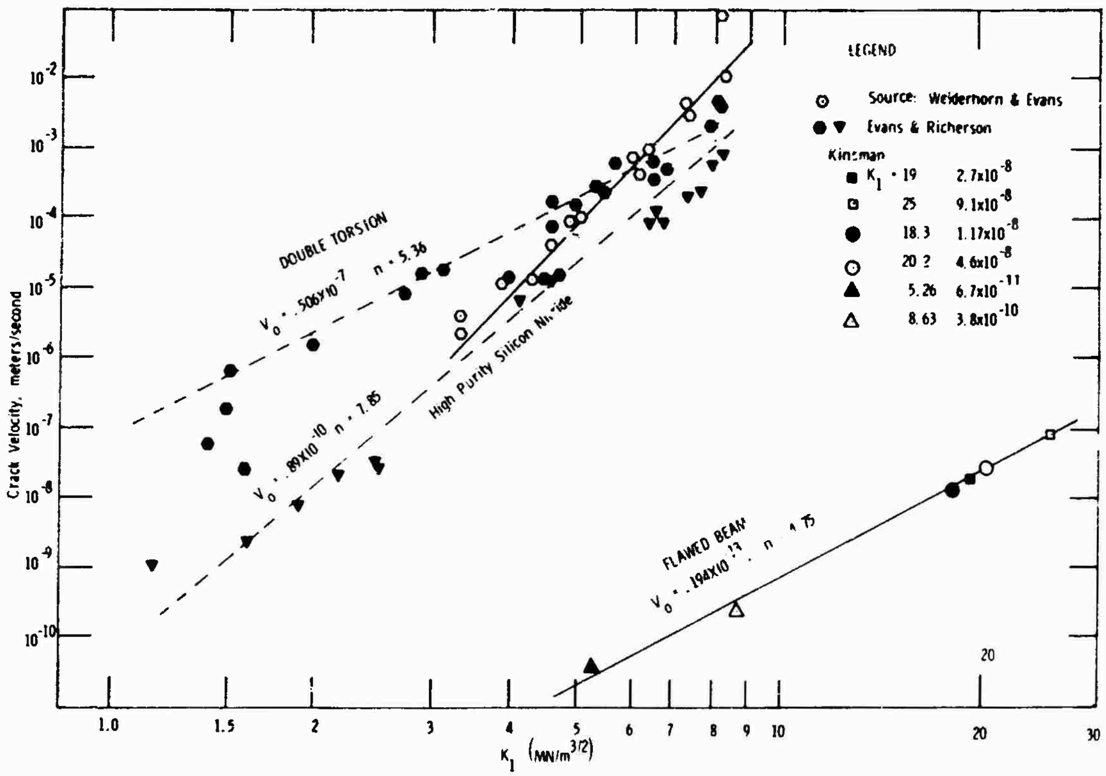


Figure 4. Summary of Available Slow Crack Growth Data for Hot-Pressed Silicon Nitride

and assuming a crack velocity versus stress intensity relation

$$\partial C/\partial t = V = K_I^n \tag{8}$$

results in the simple expression for the ratio of strengths measured at different strain rates, $\dot{\varepsilon}_1$, $\dot{\varepsilon}_2$, and for equivalent failure probabilities

$$(\sigma_1/\sigma_2)^{n+1} = \dot{\varepsilon}_1/\dot{\varepsilon}_2. \tag{9}$$

Therefore an estimate of the exponent n is available.

Alternately, under constant stress, the time to failure **T** can be estimated by use of

$$\mathbf{T} = \int_{C_i}^{C_{Ic}} dC/V. \tag{10}$$

Since

$$dC = [2K_I/(\sigma Y)^2]\, dK_I,$$
$$\mathbf{T} = 2/(\sigma Y)^2 \int_{K_{Ii}}^{K_{IC}} (K_I/V)\, dK_I \tag{11}$$

which for a simple power form of crack velocity versus stress intensity form becomes

$$T = 2K_{Ii}^{2-n}/(\sigma Y)^2\ \alpha(n-2), \tag{12}$$

for large M values, contribution of K_{IC} is neglible.

For a given batch of N specimens with initial flaw size C_j

$$T = \frac{2Y^{2-n}\sigma^{2-n}C_j^{(2-n)/2}}{(\sigma Y)^2\alpha(n-2)} = \frac{BC_j^{(2-n)/2}}{\sigma^n} \tag{13}$$

Then for specimens with the same initial flaw size and the same probability of failure

$$T\sigma^n = \alpha_1. \tag{14}$$

For an individual specimen stressed at σ_1 and failing in T_1, we now have a relationship for another specimen failing at stress σ_o in a reference time T_o. This permits construction of a nomograph, where a family of parallel lines are equi-spaced for equal logarithmic increases in failure time.[26] According to this simple theory, creep rupture data can be related to instantaneous or dynamic failures by use of Equations (9) and (14).

High temperature tension and flexure tests have been performed at various strain rates on hot-pressed silicon nitride[27] and the results are presented in Figure 5. Note in Figure 5a sufficient data was not obtained to construct a probability distribution. The indicated curves were arbitrarily taken to have the same distribution function as the intermediate strain rate (0.001 in./in./min) tests. Equation (9) was applied to this data and the results are presented in Table 1.

Using the tension data, the so-called strength-probability-time[26] nomographs in Figure 6 were prepared. The differences between these two graphs are a reflection of scatter and uncertainty in the experiments. In Figure 6a, for instance, probability of 99.99 percent is associated with lifetimes of 317 years for stress levels of 3600 psi. The corresponding values from Figure 6b are 0.32 year for 99.99 percent at 3600 psi. Equation (14)

Table 1. SUMMARY OF EXPONENT AND SPACING RATIO ESTIMATES

Data	Average Exponent n	Logarithmic Spacing Ratio
2300°F Flexure	7.29	1.329
2500°F Flexure	4.1005	1.6034
2300°F Tension (all data)	9.43	1.31
2300°F Tension (Westinghouse data)	11.93	1.20

27. McLEAN, A. F., FISHER, E. A., and BRATTON, R. J. *Brittle Materials Design, High Temperature Gas Turbine.* AMMRC CTR 72-3, CTR 72-19, CTR 73-9, CTR 73-32, CTR 74-26, CTR 74-59, Ford Motor Company, Dearborn, Mich., Contract DAAG46-71-C-0162, Army Materials and Mechanics Research Center, July 1971-December 1974.

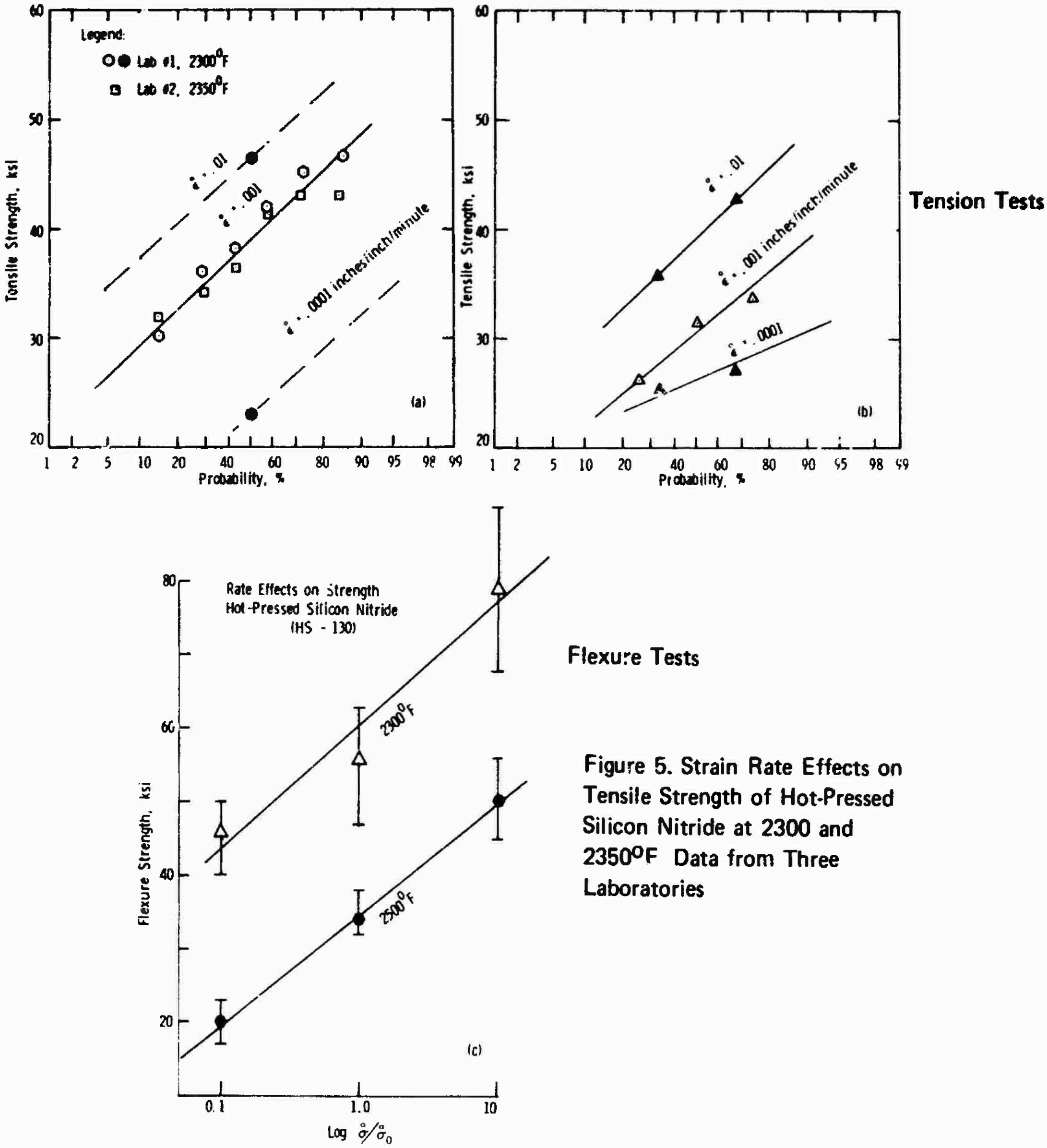


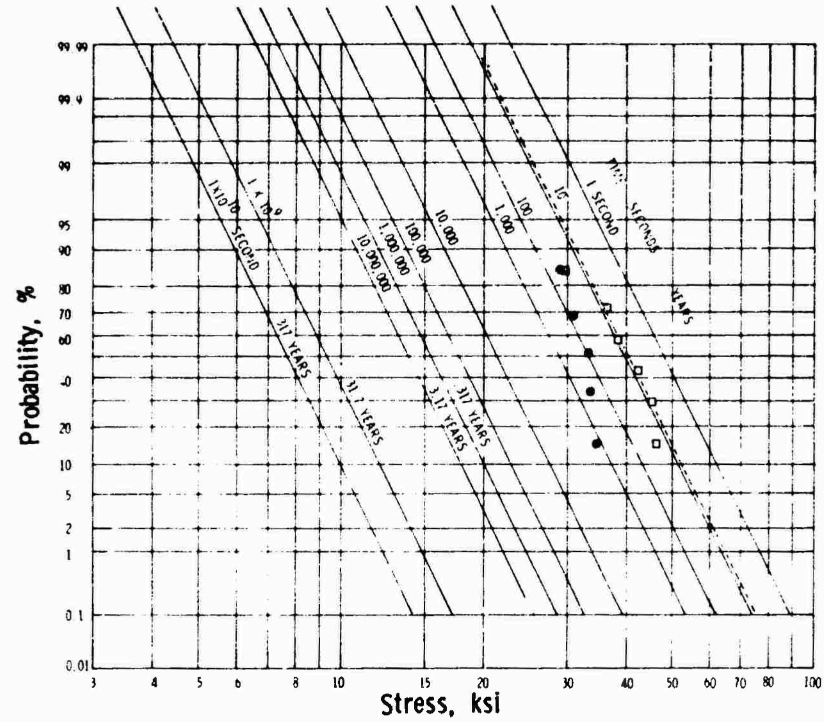
Figure 5. Strain Rate Effects on Tensile Strength of Hot-Pressed Silicon Nitride at 2300 and 2350°F Data from Three Laboratories

provides an additional check of the mentary model. Tension creep tests were performed[27] at elevated temperature on the hot-pressed (HS-130) silicon nitride. Creep rupture data is listed in Table 2.

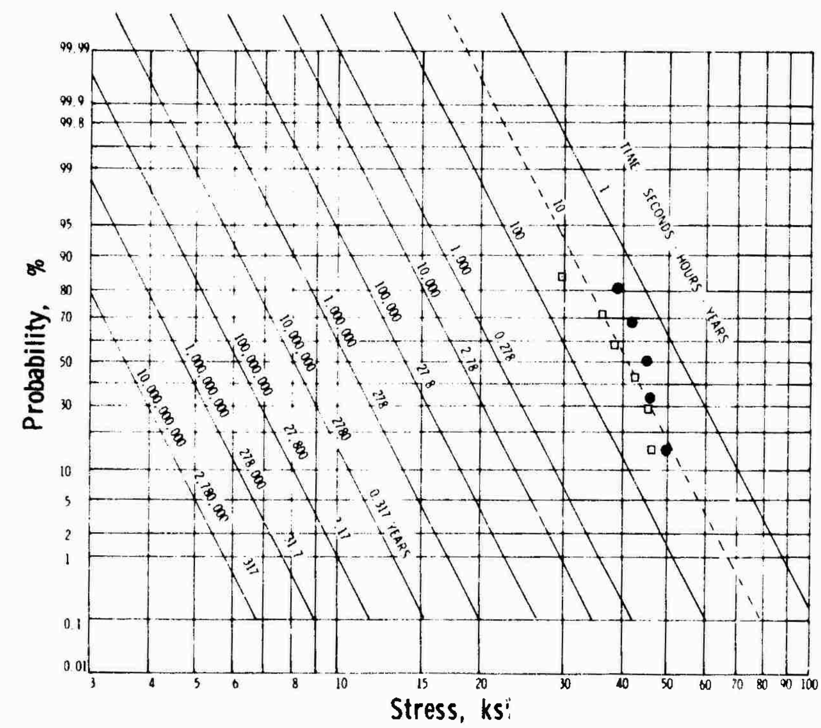
Equation (14) was applied and the creep data "normalized"[20,21,26] to 10 seconds. The transformed probability distribution is shown in Figure 6 where it is apparent that the data in Figure 6b is in better agreement with the theoretical formulation, which suggests that an n value of 9.43 is more appropriate for 2300 F behavior. The solid circles are the "transformed" creep data and should be compared to the sloping line labeled 10 seconds, or the open rectangular data points.

Table 2. TENSION CREEP AT 2300°F

Constant Stress, ksi	Failure Time, Seconds
10	4.64×10^5
10	2.16×10^6
12	1.62×10^5
12	3.17×10^5
14	6.19×10^4



a. n = 11.93, Characteristic Time τ_c = 8.8 Seconds, Ratio = 1.2



b. n = 9.43, Characteristic Time τ_c = 10 Seconds, Ratio = 1.31

Figure 6. Design Stress-Probability-Time Nomographs for Hot-Pressed Silicon Nitride at 2300°F

ALTERNATE LIFE ESTIMATING PROCEDURES

We have seen that various test methods have evolved whereby the crack velocity V in a specimen can be recorded simply in terms of nominal stress intensity K_I. Crack growth in terms of the K-V relations can be theoretically related to an initial flaw size C. For example, application of a stress σ produces a stress intensity factor $K_I = Y\sigma(C)^{1/2}$ corresponding to a crack velocity V. If C increases, the corresponding K_I will increase. Therefore, when K_I reaches a particular critical value K_{IC}, or some lesser value depending on environmental conditions, catastrophic failure occurs.

For constant σ in a delayed fracture test the time to failure is given

$$T = \int_{C_i}^{C_{I_c}} (dC/V) \tag{15}$$

with a grossly simplifying assumption of a known average initial flaw size and where

$$C_i = \text{initial crack size}$$

$$C_{I_i} = \text{critical crack size,}$$

since

$$dc = (2K_I/(\sigma Y)^2)dK_I$$

then

$$T = 2/(\sigma Y)^2 \int_{K_I'}^{K_I''} (K_I/V)\,dK_I \tag{16}$$

where limits of integration are determined from the K-V diagram.

Thus far it is evident that a fairly large uncertainty exists as to proper choice of the K-V data. However, let us assume that a particular set of data has been chosen as acceptable. Then several decisions must be made in order to proceed even with the deterministic and direct integration of Equation (15). The first question relates to analytical representation of the stress intensity-crack velocity function, whereas the second concern is the range in values of the integration. Let us first consider an analytical formulation.

A commonly used functional relationship for V is the so-called "power law",

$$V = A_o K_I^m \tag{17}$$

where A_o and m are constants determined from a least-squares fit of data from typical K-V diagrams (Figure 4). Making appropriate substitutions, T may be defined as

$$T = 2/\left((m-2)(\sigma Y)^2 A_0\right) \left[K_I'^{(2-m)} - K_I''^{(2-m)}\right] \tag{18}$$

An exponent form $V = V_o^{mK_I}$ has also been used in a similar manner.

Alternatively, if time to failure is defined as

$$T = 2/(\sigma Y)^2 \int_{K_I'}^{K_I''} F \, dK_I \tag{19}$$

where $F = K_I/V$, then a plot of log F versus K_I reveals a linear relationship, see Figure 7.

Taking advantage of this fact one can relate $F(K_I)$ as

$$\log F = A_1 K_I + B_1 \text{ or}$$
$$F = Ae^{BK_I}. \tag{20}$$

Now a least-squares fit of data from F versus K_I data determines A and B and can be written as

$$T = \frac{2}{(\sigma Y)^2} \int_{K_I'}^{K_I''} Ae^{BK_I} \, dK_I \text{ or}$$
$$T = \frac{2}{(\sigma Y)^2} \left[\frac{A}{B}\left(e^{BK_I''} - e^{BK'_I}\right)\right] \tag{21}$$

The above form for F is used in analyzing crack growth data presented in this paper.

Using $F(K_I)$ instead of power law (V) reduces values of the exponent in the fractional form (K_I/V). This is an important consideration because large exponent values ($n \geq 10$) result in an unstable T value when a large variance in K_I is applied, see Figure 8.

Available silicon nitride data and the limited slow crack growth results for glass examined in this study have tended to follow this linear relation, whereas with separate representations for crack velocity and stress intensity of the form $F = K_I/V$ gends to depart from this apparent linear response, see Figure 9. Therefore it seems more advantageous to fit $F(K_I)$ data directly instead of fitting $V(K_I)$ data and then defining integrand as K_I/V.

In fitting the data it is not necessary to obtain a fit over the entire range of K_I. For the data examined, it was usually possible to consider only three orders of magnitude change in V. That is, if initially $V = 1\times10^{-10}$, the range of K_I is selected such that V is in the vicinity of 1×10^{-7}. This is possible because the contributions to lifetime T are negligible beyond the determined range, see

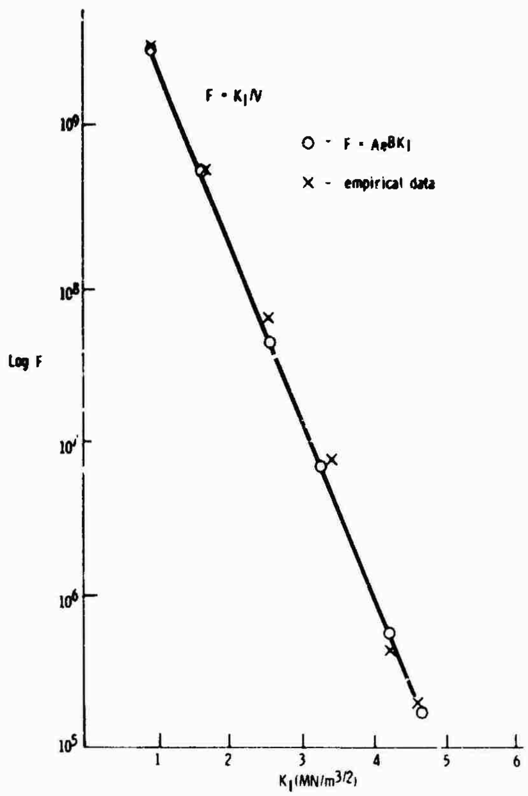


Figure 7. Comparison of Exponential Fit for Combined Integrand - High Purity Data for Silicon Nitride

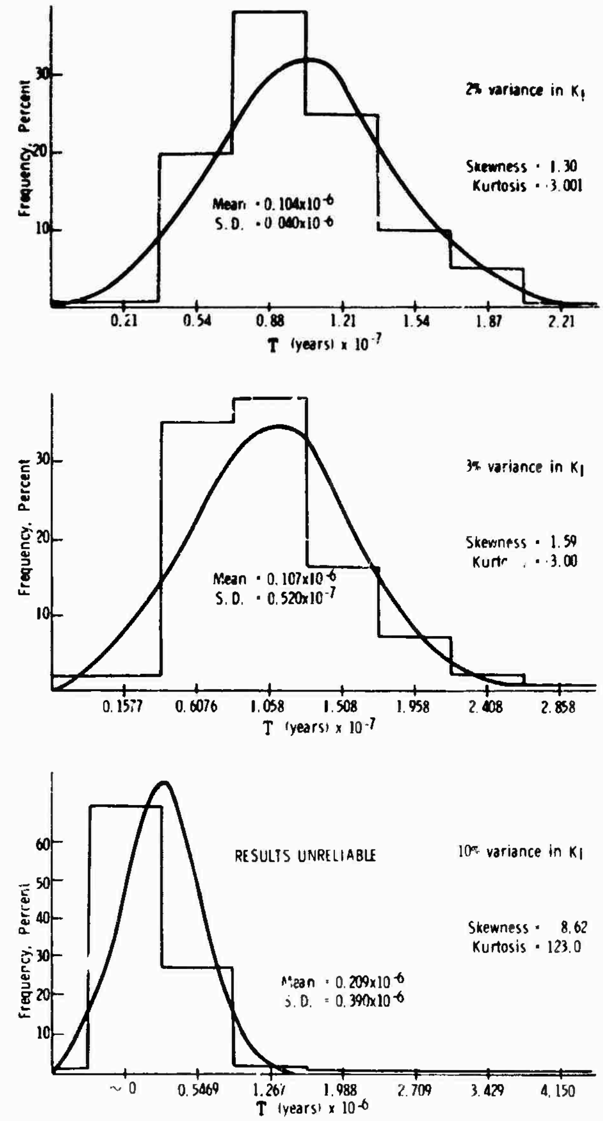


Figure 8. Illustrating Sensitivity of Life Estimate to Variance in Nominal Stress Intensity K_I. σ = 20 ksi

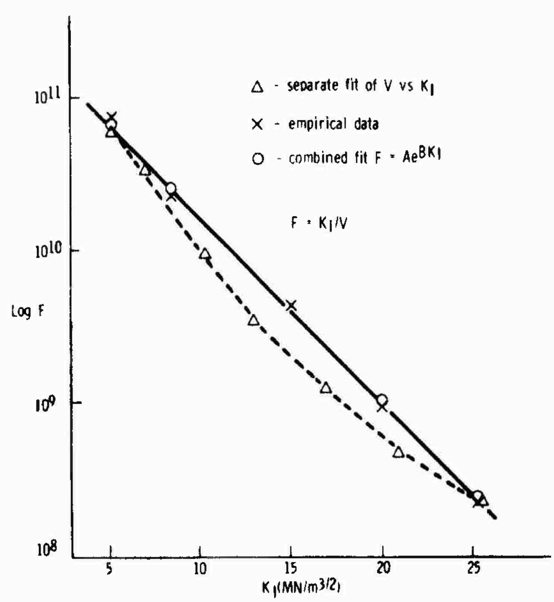


Figure 9. Comparison of Integrand Fitting Techniques. Ln F versus K_I Flawed Beam (after Kinsman, Ref. 19)

Table 3. It may be noted that for each order of magnitude change a significant digit is obtained for T. This minimizes the need for a large number of data points in the experimentation, at least for the lifetime estimate.

An alternate method, using a polynomial representation in place of the other functional forms, was examined to obtain more flexibility in the curve-fitting procedure. A three-point Lagrangian interpolation scheme proved to be successful. This interpolation process combined with a piecewise integration scheme provides an acceptable method of obtaining T when the standard functional representations are not acceptable. This method is also adaptable for use with the Monte Carlo method as shown in Figure 10.

The method involves initially selecting a polynomial form

$$F = C_1 K_I^2 + C_2 K_I + C_3 \tag{22}$$

and fitting three points at a time to F versus K_I data using the Lagrangian process.

Secondly, the time segment T_2 for the first two points is defined as

$$T_2 = \int_{K_{I_1}}^{K_{I_2}} (C_1 K_I^2 + C_2 K_I + C_3)\, dK_I \tag{23}$$

where K_{I_1}, K_{I_2} = first two data values for K_I. The process is repeated N times until desirable convergence for failure time estimate is observed. In general

$$T_{(i+1)} = \int_{K_{I_i}}^{K_{I(i+1)}} (C_1 K_I^2 + C_2 K_I + C_3)\, dK_I, \tag{24}$$

Table 3. FLAW BEAM DATA (Piecewise Integration)

$$T_{i+1} = \int_{K_{I_i}}^{K_{I_{i+1}}} (C_1 K_I^2 + C_2 K_I + C_3) dK_I$$

σ = 10 ksi

i	$K_{I_{i+1}}$	K_{I_i}	T_{i+1} (days)
1	8.63	5.26	200
2	18.3	8.63	114
3	20.2	18.30	3
		$T = \sum_{i=1}^{3} T_{i+1}$	= 317

T*(indicates determination over entire range of data using $F = Ae^{BK_I}$.) = 315

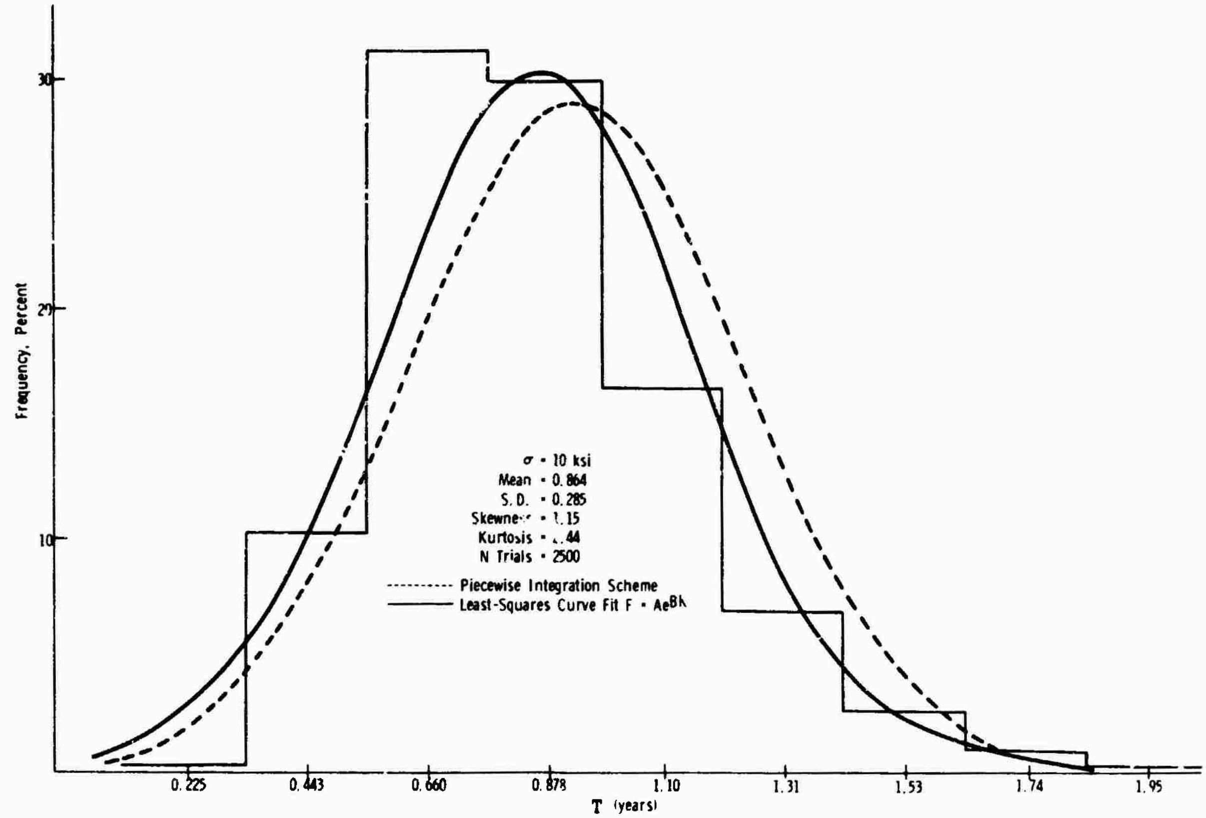


Figure 10. Comparison of Polynomial and Piecewise Integration for Life Estimates, Based on Flawed Beam Data, Si_3N_4

and

$$T \approx \sum_{i=1}^{N} T_{(i+1)}$$

where C_i's are determined from curve-fitting procedures for each segment. These various formulation were treated both in deterministic ways as well as by use of digital simulation techniques, which are described next.

MONTE CARLO METHOD

The Monte Carlo method is a desirable means of determining time to failure for slow crack growth since the controlling parameters, e.g., K_I, V, Y, and σ are inherently variable. The relative complexity of the functional relationship, in addition to the expense of extensive experimentation, further suggest the need for the Monte Carlo method. The method avoids restrictions on the exact specification of the statistical distributions of the variables since simple approximating functions can serve well in the scheme. In general, this prevents formulation of incorrect frequency distributions for the quantities of interest.

In this paper determination of lifetime estimates T by the Monte Carlo method involves representing each of the independent variables by a set of normally distributed random numbers. Use of normal distributions is not a requirement. However, due to resulting simplifications, normal distributions were used in this initial phase of our work. The pertinent equation, including crack geometry factor, commonly applied for lifetime estimates is:

$$T = 2/(\sigma Y)^2 \int_{K_I'}^{K_I''} (K_I/V)\, dK_I \tag{25}$$

where σ = applied stress

Y = parameter depending on appropriate crack geometry

K_I', K_I'' = stress intensity (initial and final)

K_I = stress intensity

V = crack velocity

A value is specified for each variable by choosing a number at random from its corresponding distribution. The resulting value for T is determined for each set of randomly selected variables. The computation is repeated and a probability distribution is obtained for the defect sizes.

The normally distributed random numbers were generated by a Univac 1108 subroutine. The procedure is simply one of generating uniform random numbers and solving for X in the relation

$$\int_{-\infty}^{X} f_i \, dX = R \tag{26}$$

where R = uniform random number and

f_i = normal frequency distribution.

As noted previously, if the probability distributions of the controlling variables are known from some experimental results or from an analytical basis, then the appropriate distribution function f_i may be used.

RESULTS AND DISCUSSION

The Monte Carlo simulation scheme is a good method for analyzing slow crack growth data to obtain acceptable time to failure results. This is particularly true because of the experimental errors associated with V-K_I determinations. The uncertainty of a theoretical basis for the power law or other approximating function to represent data in the lifetime determination provides an additional motivation for using the method.

With regard to application of the Monte Carlo method, we observed that selection of the appropriate number of simulations must rely on considering third and fourth moments of the resulting statistical distribution. It was also observed

that mean values and standard deviations were fairly constant after a relatively few trials, whereas the third and fourth moments continued to exhibit instability until considerably larger numbers of simulations had been completed. The number of cells in histograms exhibited in Figures 8 and 10 were determined from Sturgis' equation $NG = 1 + 3.3 \log_{10}(N)$, where N is number of trials and NG number of cells.

Use of Wilkins' glass data in Figure 8 shows the gross effect of varying K_I by 10 percent as compared with a 2 percent variation. This sensitivity results from using exponent values that are quite large in the relationship $F = B \exp (AK_I)$ in the curve-fitting process. As an example, for Wilkins' results A = -22.9 and $B = 0.144 \times 10^{12}$, while high purity data produced values of A = -2.64 and $B = 0.370 \times 10^{11}$. The large exponent values result from the rapid change in crack velocity with respect to change in K_I. The more brittle materials have this characteristic of high exponential values.

From examination of Figure 8, one may conclude that if error measure is greater than 2 percent in K_I, the result is a distorted account of the time to failure. Such a material would be highly unreliable for structural purposes.

In Figure 9 the effect of using separate fits for $V = V_o K_I^m$ and then applying them to the integrand $F = K_I/V$ is quite obvious. This weighting of the junction $F = K_I/V$ distorts representation of the integrand in lifetime estimate integral. For this case there is a highly conservative estimate of time. In determining a separate functional relationship for V, one is essentially considering the integrand $F = K_I/V$ in a manner that assumes K_I a constant. Weighting of F by separate fitting of V is not necessary if one utilizes the F versus K_I curve shown in Figure 9. It is quite fortunate that for all data analyzed (see Figure 9), log F versus K_I results in a linear plot. This allows for the simple representation $F = A_o e^{MK_I}$ to be used in the analysis.

As a final remark regarding Table 3, it seems with rapid order of magnitude changes in V with respect K_I one should take advantage of this fact and not attempt to obtain data beyond that necessary to calculate T accurately to three significant digits. This will also eliminate the need for curve fitting additional data and in some instances results in increasing the exponential value m.

A number of investigators have espoused the use of proof testing in conjunction with slow crack growth concepts.[28-30] Unfortunately, the conventional application of such proof test concepts generally involves backward extrapolation of the crack velocity data into time regimes which are exceedingly difficult to define experimentally. In addition, the data have ordinarily been analyzed with a power law form, which is a least-squares fit over the entire range of observation. Such procedures perhaps overweigh the trends exhibited by the larger values of K_I, which in reality contribute little to overall life. A more reasonable procedure might be to consider a shorter range of K_I, closer to the region of extrapolation. In any case it is well worth noting the actual uncertainty introduced by use of proof testing. Furthermore, the estimating method is extremely sensitive to values

28. WIEDERHORN, S. M. *Reliability, Life Prediction and Proof Testing of Ceramics.* NBSIR 74-486, National Bureau of Standards, Washington, D. C., May 1974.
29. EVANS, A. G., WIEDERHORN, S. M., LINZER, M., and FULLER, E. R. Jr. *The Proof Testing of Porcelain Insulators and the Application of Acoustic Emission.* NBSIR 74-512, National Bureau of Standards, Washington, D. C., 1974.
30. EVANS, A. G. *Understanding the Characteristics of Brittle Materials.* ASME Paper 75-GT-87. March 1975.

determined by such extrapolation, and care must be taken in selecting the proof stress ratio. Examination of Wilkins' data[6] is indicative of potential disastrous effects of conventional proof test concepts. For instance, the remark that differences of b of 7 or 8 percent in $V = V_o K_I^b$, produces a four- to fivefold difference in T_{min} is not correct. If one evaluates T_{min} at initial K_I value rather than extrapolates, the maximum difference in T_{min} is singlefold. This oversight seems to be fairly common among other researchers in ceramics.

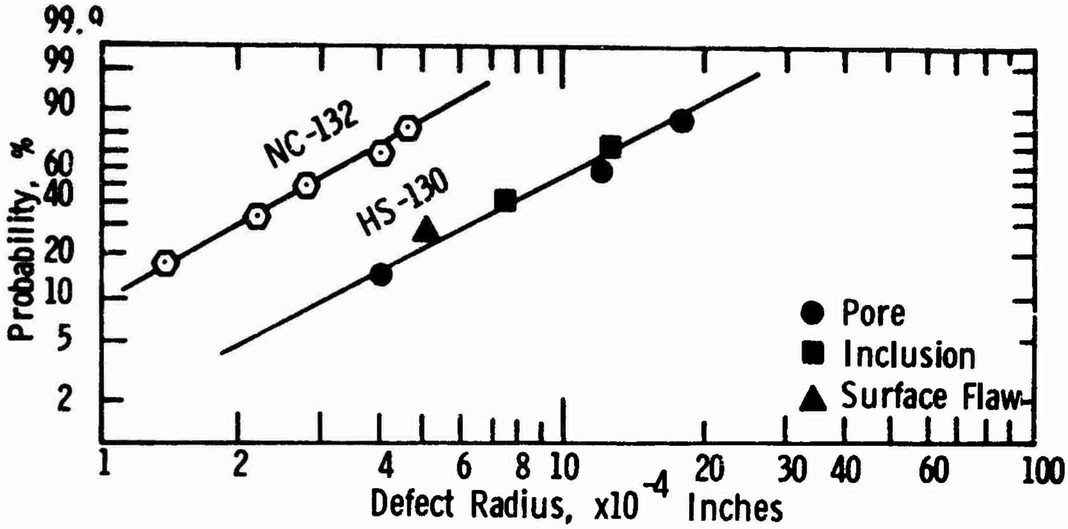
SUMMARY AND CONCLUSIONS

Review of the literature has shown that further improvements are desirable in theoretical analysis of subcritical crack growth specimens commonly used for ceramic materials. Available data for hot-pressed silicon nitride was relatively scarce and the results suggested considerable variation of observed slow crack growth data. Scatter in the data is partly inherent, but is probably also related to variations of controlling impurity contents. It appears that crack velocity observations differed depending on whether they were made on a macro- versus a microscale.

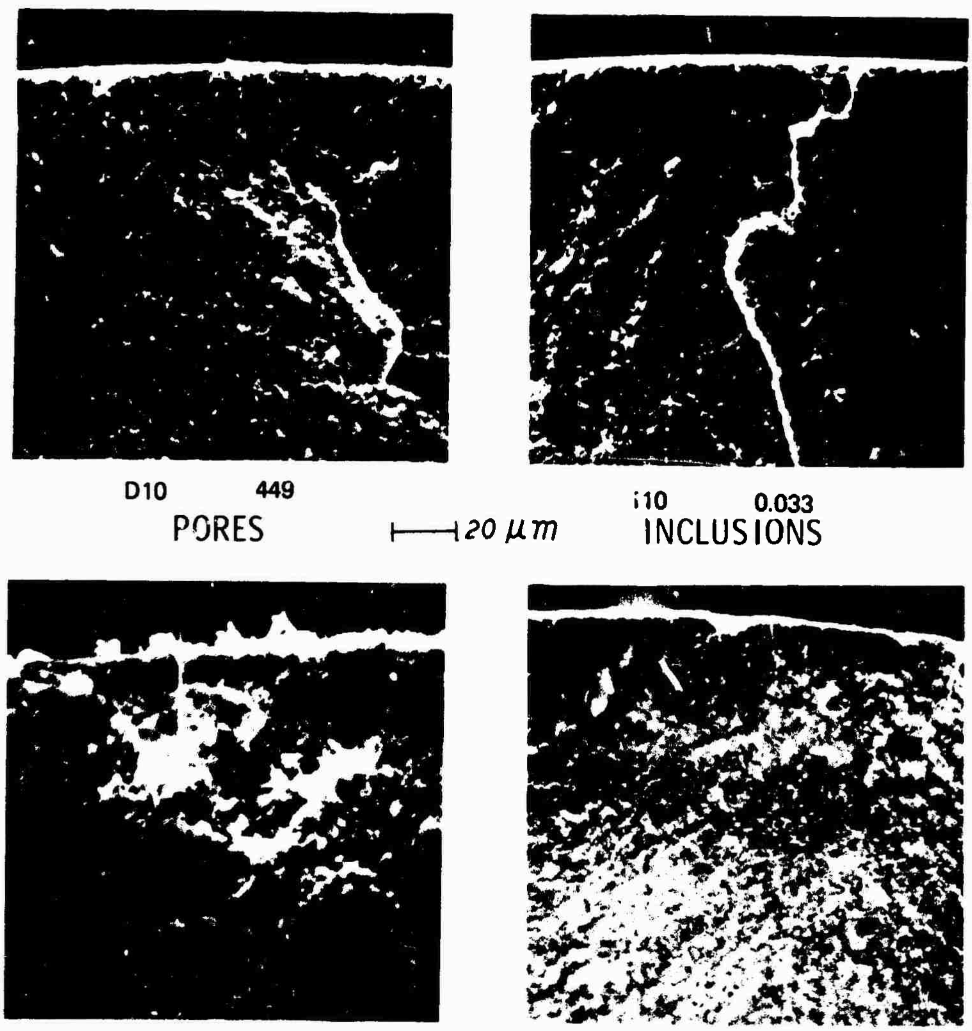
A key question relates to the determination of the initial flaw size. Ordinary concepts utilize the notion of an average critical flaw size as the starting point. In this context it is interesting to refer to Figure 11, which illustrates typical critical flaws and their distribution as observed by fractography of mechanical properties test specimens.[31-32] It is also worth observing that in approximately 50 percent of the specimens examined, critical flaws could not be identified via fracture mirror observation, suggesting the existence of rather small defects. In principle,[33] the slow crack growth equation could be integrated where distribution of initial flaws and their subsequent growth would be taken into account. In reality, the computations are exceedingly difficult, and the matter of whether the crack velocity versus stress intensity relationship is influenced by the area of the crack and the nature of the initial flaw distribution would probably be more easily investigated experimentally.

With reference to the different procedures for estimating life, consider the results shown in Figure 12 which presents the cumulative distribution function based on tension tests conducted at different strain rates; Figures 13 and 14 illustrate results of the Monte Carlo computation for the different slow crack growth data. The range in mean value of survival time is shown by comparing Figure 12 near the 40 percent probability level (∿ 0.175 year) with Figure 13a (∿ 0.864 year) and Figure 14 (∿ 0.0047 year). These life estimates exhibit the uncertainties introduced via test procedures, analytical technique, and materials impurity. Figure 13 also indicates the influence of design stress (2, 10, and 20 ksi), for 10 percent variance in all controlling parameters.

31. GRUVER, R. M., SOTTER, W. A., and KIRCHNER, H. P. *Fractography of Ceramics.* Ceramic Finishing Company, State College, Pa., Naval Air Systems Command Contract N00019-73-C-0356, November 1974.
32. BARATTA, F. I., DRISCOLL, G. W., and KATZ, R. N. *The Use of Fracture Mechanics and Fractography to Define Surface Requirements for Si_3N_4.* In Ceramics for High Performance Applications, J. J. Burke, A. E. Gorum, and R. N. Katz, Eds., Brook Hill Publishing Company, Chestnut Hill, Mass., 1975, p. 445-476.
33. FREUDENTHAL, A. M. *New Aspects of Fatigue and Fracture Mechanics.* The George Washington University, Washington, D. C., Office of Naval Research Contract N00014-67-A-0214-0011, December 1973.



PENETRATION FLAWS



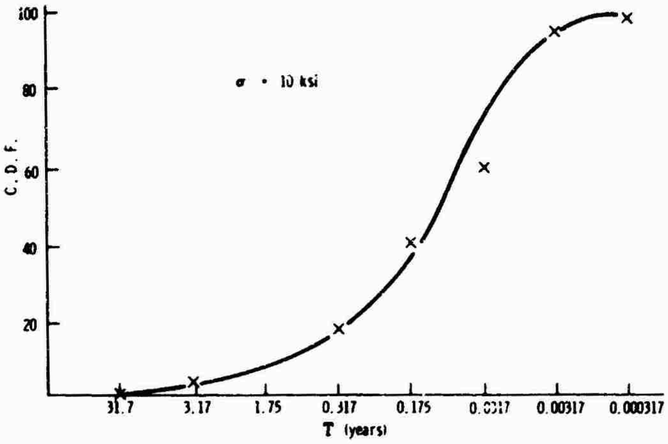


Figure 12. Cumulative Distribution Function for Life Estimate via High Strain Rate Tests, 10 ksi Design Stress, HS-130 Silicon Nitride at 2300°F

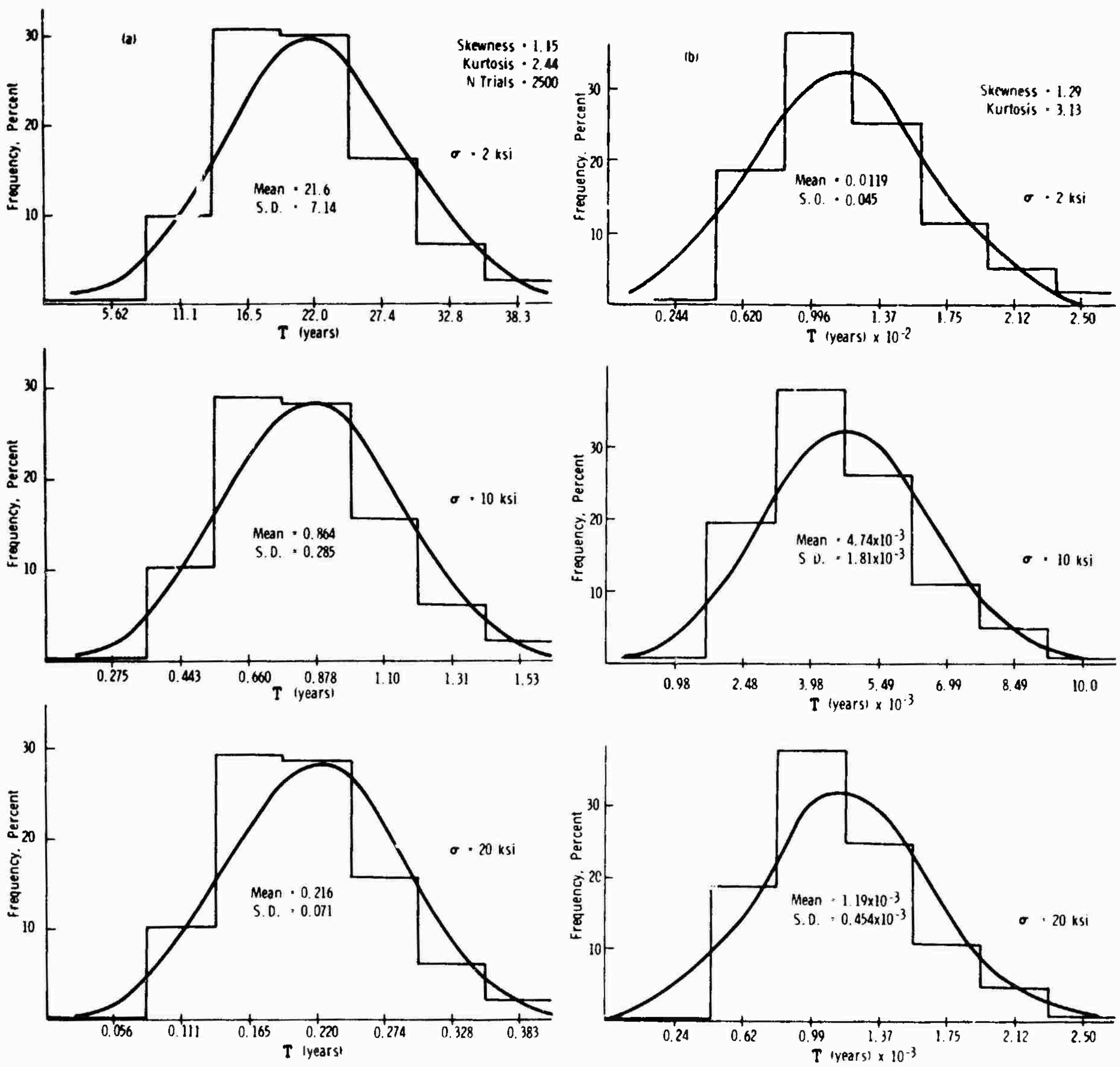


Figure 13. Life Estimates Based on Flawed Beam and Double Torsion Tests, Commercial and High Purity Silicon Nitride, 10% Variance in Parameters at 2500° F

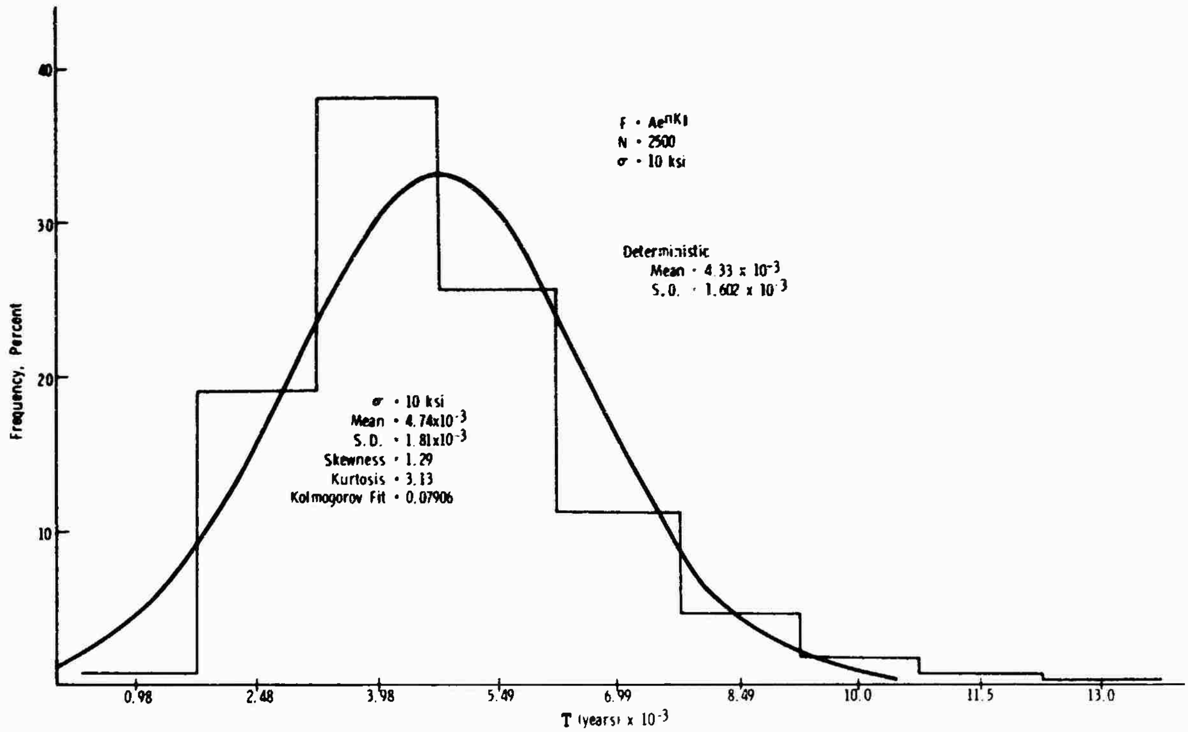


Figure 14. Distribution of Life Estimates for 10 ksi Design Stress, 10% Variance in Controlling Parameters, Double Torsion Test, High Purity Silicon Nitride at 2500°F

With regard to choice of the various life estimating techniques, we see that the strength-probability-time nomograph (Figure 6) compared with the flawed beam results (Figure 13) provides a conservative estimate, ignoring the temperature difference. Computations for the commercial Norton HS-130 material, based on the double torsion tests (see Figure 4), provide an even shorter life estimate. It would appear reasonable to use the high strain rate data, provided that the nominal surface finish and volume of the test specimen is similar to the dimension of the structural component of interest. The choice of double torsion versus flawed beam data must be resolved by further study. In conclusion, it is evident that an improved fracture and strength data base is required and further refinements of analysis will be necessary to substantiate or refute the opinions presented in this preliminary assessment.

ACKNOWLEDGMENTS

The support of the Advanced Research Projects Agency is gratefully acknowledged and the authors wish especially to thank Dr. E. C. Van Reuth of ARPA for his support of this study. Further acknowledgment must be extended to Cynthia C. Baird for numerous calculations and preparation of the illustrations.